

Docket No.: 0599-0219PUS1

(PATENT)

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

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In re Patent Application of:  
Shunichi OSADA et al.

Application No.: 10/594,593

Confirmation No.: 6807

Filed: December 19, 2006

Art Unit: 1794

For: MULTILAYER FILM

Examiner: P. J. Khatri

**DECLARATION UNDER 37 CFR 1.132**

Commissioner for Patents

P.O. Box 1450

Alexandria, VA 22313-1450

Sir:

I, Syunichi Osada, residing at 10-28, Nishikori 3chome, Otsu-shi, Otsu, 520 Japan, do declare and state as follows:

1. I am a co-inventor of this invention disclosed in above-identified U.S. application and hence I am fully familiar therewith.

2. I am an engineer having earned a master's degree of engineering from the Gifu university graduate school of engineering in March 1995 and have been employed by TORAY INDUSTRIES INC. of 1-1, Nihonbashi-Muromachi 2-chome, Chuo-ku, Tokyo, 103 Japan since April of 1995. I have been engaged mainly in research and development on multilayer film and polyester film manufacturing processes.

3. I have reviewed the office action dated December 30, 2010 as well as Hebrink (US 2001-0019182). In order to show the differences in properties and structure between a film obtained by a process similar to Hebrink and the film obtained by the method of the present invention, I hereby respectfully submit a description of comparative experimental data based on experimental tests that I conducted several years ago.

4. Object of Experiments:

To clarify the differences between the film obtained by the process of Hebrink and the film obtained by the method of the present invention.

5. Site of Experiments:

Films and Films Products Research Laboratories, TORAY INDUSTRIES, INC.,  
1-1, Sonoyama 1-chome, Otsu-shi, Shiga, JAPAN

6. Person in Charge of Experiments:

Syunichi OSADA

7. Duration of Experiments:

From October 14, 2004 to January 28, 2005

## 8. Methods of Experiments:

### Experiment (1)

As two types of thermoplastic resins, Thermoplastic Resin A and Thermoplastic Resin B were prepared. As the Thermoplastic Resin A, a polyethylene terephthalate (PET) having an intrinsic viscosity of 0.65 (Type "F20S" produced by TORAY INDUSTRIES, INC.) was used. This Thermoplastic Resin A was a crystalline resin. As the Thermoplastic Resin B, a resin produced by kneading and alloying a polyethylene terephthalate in which cyclohexane dimethanol in an amount of 30 mol% was copolymerized with ethylene glycol (CHDM-copolymerized PET) ("PETG6763" produced by EASTMAN) and a polyethylene terephthalate having an intrinsic viscosity of 0.65 at a weight ratio of 85:15 in a biaxial extruder was used. The polyethylene terephthalate in which cyclohexane dimethanol in an amount of 30 mol% was copolymerized with ethylene glycol (CHDM-copolymerized PET) (PETG6763 produced by EASTMAN) was a non-crystalline resin and the polyethylene terephthalate having an intrinsic viscosity of 0.65 was a crystalline resin. The density of the Thermoplastic Resin A before drying was  $1.336 \text{ g/cm}^3$  and the density of the Thermoplastic Resin B before drying was  $1.285 \text{ g/cm}^3$ . These Thermoplastic Resin A and Thermoplastic Resin B were supplied to an extruder, respectively, after drying.

The Thermoplastic Resin A and Thermoplastic Resin B were melted at  $280^\circ\text{C}$  in the extruder, respectively, and the resins were merged using a feed block giving 201 layers, after passing through a gear pump and a filter. The feed block giving 201 layers was composed of only one slit member having 201 slits. The thicknesses of the layers in the merged Thermoplastic Resin A and Thermoplastic Resin B were conditioned such that the thickness of each layer was increased from the side of a surface to the side of the opposite surface (slope pattern) and 101 layers of the Thermoplastic Resin A and 100 layers of the Thermoplastic Resin B were alternately laminated. As for the shape of the slits, each slit had a length of 40 mm and the ratio

of (slit area at the side in which the resin is not introduced)/(slit area at the side in which the resin is introduced) was set to 0.5. The thickness of each layer was adjusted by the shape of a slit (formed with a precision of processing of 0.01 mm) provided in the channel for each layer in the feed block. The laminate was formed such that the both outermost sides of the laminate were made of Thermoplastic Resin A. The shape of the feed block and the extrusion amount were adjusted such that the thickness ratio of adjacent A layer and B layer (thickness of A layer / thickness of B layer) was 0.95. The thus obtained laminate having totally 201 layers was guided to two-tiered static mixers called a square mixer to form a laminate having totally 801 layers (the adjacent layers made of the same resin were counted as one layer). The resulting laminate was supplied to a die and shaped into the form of a sheet, followed by rapidly cooling the obtained sheet on a casting drum whose surface temperature was kept at 25°C while applying electrostatic force.

After heating the obtained cast film with a group of rolls set at 68°C to 82°C, the cast film was stretched in the longitudinal direction at a draw ratio of 3.3 times and the resulting film was once cooled. Subsequently corona discharge treatment was processed on the both surfaces of the resulting uniaxially oriented film in the air to attain a wetting tension of the base film of 55 mN/m, and a laminate layer-forming coating solution composed of (a polyester resin having a glass transition temperature of 18°C)/(a polyester resin having a glass transition temperature of 82°C)/silica particles with an average particle size of 100 nm was coated on the treated surfaces, thereby forming transparent, lubricating and adhesive layers.

The thus obtained uniaxially oriented film was introduced into a tenter and stretched in the transverse direction at 110°C at a draw ratio of 3.5 times, after preliminary heating with hot air at 100°C. The stretched film was heat-treated in the tenter with hot air at 230°C, and then relaxed by 5% in the transverse direction at the same temperature. The film was then allowed to slowly cool to room temperature, and

cure. The obtained film had a thickness of 120  $\mu\text{m}$ . The obtained results are shown in Table 1. With the film obtained in this experiment, although the changes in the optical properties by heating were scarcely observed, irregularity in lamination was large so that reflection efficiency of the near infrared radiation was low, and reflection in the visible light region was observed. Thus, a near infrared with poor performance was resulted. Further, variation in the reflectance in the transverse direction was large, and a filter having a uniform reflectance was not obtained.

### Experiment (2)

As two types of thermoplastic resins, Thermoplastic Resin A and Thermoplastic Resin B were prepared. As the Thermoplastic Resin A, polyethylene terephthalate (PET) having an intrinsic viscosity of 0.65 (Type "F20S" produced by TORAY INDUSTRIES, INC.) was used. This Thermoplastic Resin A was a crystalline resin. As the Thermoplastic Resin B, a resin produced by kneading and alloying a polyethylene terephthalate in which cyclohexane dimethanol in an amount of 30 mol% was copolymerized with ethylene glycol (CHDM-copolymerized PET) ("PETG6763" produced by EASTMAN) and a polyethylene terephthalate having an intrinsic viscosity of 0.65 at a weight ratio of 85:15 in a biaxial extruder was used. The polyethylene terephthalate in which cyclohexane dimethanol in an amount of 30 mol% was copolymerized with ethylene glycol (CHDM-copolymerized PET) ("PETG6763" produced by EASTMAN) was a non-crystalline resin and the polyethylene terephthalate having an intrinsic viscosity of 0.65 was a crystalline resin. The density of the Thermoplastic Resin A before drying was  $1.336 \text{ g/cm}^3$  and the density of the Thermoplastic Resin B before drying was  $1.285 \text{ g/cm}^3$ . These Thermoplastic Resin A and Thermoplastic Resin B were supplied to an extruder, respectively, after drying.

The Thermoplastic Resin A and Thermoplastic Resin B were melted at  $280^\circ\text{C}$  in the extruder, respectively, and the resins were merged using a feed block giving 801

layers, after passing through a gear pump and a filter. As the feed block giving 801 layers, the apparatus as shown in Figs. 1 and 4 in this present application was used. The feed block was composed of three slit members each having 267 microscopic slits. The thicknesses of the layers of the merged Thermoplastic Resin A and Thermoplastic Resin B were conditioned such that the thickness of each layer was increased from the side of a surface to the side of the opposite surface (slope pattern) and 401 layers of the Thermoplastic Resin A and 400 layers of the Thermoplastic Resin B were alternately laminated. As for the shape of the slits, each slit had a length of 40 mm and the ratio of (slit area at the side in which the resin is not introduced)/(slit area at the side in which the resin is introduced) was set to 0.5. The thickness of each layer was adjusted by the shape of a slit (formed with a precision of processing of 0.01 mm) provided in the channel for each layer in the feed block. The laminate was formed such that the both outermost sides of the laminate were made of the Thermoplastic Resin A. The shape of the feed block and the extrusion amount were adjusted such that the thickness ratio of adjacent A layer and B layer (thickness of A layer / thickness of B layer) was 0.95. The obtained laminate having totally 801 layers was supplied to a die and shaped into the form of a sheet, followed by rapidly cooling the obtained sheet on a casting drum whose surface temperature was kept at 25°C while applying electrostatic force.

After heating the obtained cast film with a group of rolls set at 68°C to 82°C, the cast film was stretched in the longitudinal direction at a draw ratio of 3.3 times, and the resulting film was once cooled. Subsequently, corona discharge treatment was processed on the both surfaces of the resulting uniaxially oriented film in the air to attain a wetting tension of the base film of 55 mN/m, and a laminate layer-forming coating solution composed of (a polyester resin having a glass transition temperature of 18°C)/(a polyester resin having a glass transition temperature of 82°C)/silica particles with an average particle size of 100 nm was coated on the treated surfaces, thereby forming transparent, lubricating and adhesive layers.

The thus obtained uniaxially oriented film was introduced into a tenter and stretched in the transverse direction at 110°C at a draw ratio of 3.5 times after preliminary heating at 100°C. The stretched film was heat-treated as it is in the tenter with hot air at 230°C, and then relaxed by 5% in the transverse direction at the same temperature. The film was then allowed to slowly cool to room temperature, and cure. The obtained film had a thickness of 120  $\mu\text{m}$ . The obtained results are shown in Table 1. In this resulted film, changes in the optical properties under heating were scarcely observed. And a colorless transparent near infrared filter which efficiently reflects near infrared radiation and yet the higher order reflection in the visible light region was scarcely observed was resulted. Since the irregularity in lamination was smaller than that of the film obtained in above Experiment 1), a near infrared filter with a high efficiency was obtained.

#### 9. Results:

The results of evaluations of the films are summarized in Table 1.

Table 1

		Experiment 1	Experiment 2
Layer Structure	Thermoplastic Resin A	PET	PET
	Thermoplastic Resin B	CHDM-copolymerized PET + PET	CHDM-copolymerized PET + PET
	Number of Laminated Layers	801	801
	Thickness Ratio of Adjacent A Layer to B layer	0.95	0.95
Optical Properties before Heating	Reflectance at Peak of Reflection (R1 (%))	45	90
	Wavelength at Peak of Reflection (nm)	950-1450	800-1250
	Reflectance in Secondary Reflection Band (%)	39	11
	Wavelength in Secondary Reflection Band (nm)	475-725	400-625
	Difference in Reflectance in Transverse Direction (%)	20	8
Optical Properties after Heating	Reflectance at Peak of Reflection (R2 (%))	45	90
	R1 - R2 (%)	0	0
Detachment Test	Number of Detached Lattices	0	0
Precision of Lamination	Irregularity in Lamination (%)	42	14
Ratio of Thermal Contraction	Longitudinal Direction (%)	0.2	0.2
	Transverse Direction (%)	0.2	0.2



## 10. Conclusion:

From the results of these Experiments, I conclude that by employing the feedblock used in the present invention, i.e. a feedblock that separately includes at least two or more members having a number of microscopic slits, a film is obtained which has a smaller irregularities in lamination and smaller variations in the reflectance in the transverse direction than that obtained by employing the combination of a smaller feedblock and a "square mixer" corresponding to the "multipulier" used in Hebrink. I also note that the feedblock and square mixer used in Experiment (1) are much closer to the embodiment of Experiment (2) than any of the examples of Hebrink.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

31, March, 2010

Date

Syunichi Osada.

(Syunichi Osada)